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EG&G - ROCKY FLATS PLANT
ENVIRONMENTAL MANAGEMENT

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**ROCKY FLATS PLANT
EMD OPERATING
PROCEDURES MANUAL**

**Manual No.: 5-21000-OPS-SW
Procedure No. Table of Contents, Rev 4
Page 1 of 2
Effective Date. 05/12/92
Organization: Environmental Management**

THIS IS ONE VOLUME OF A SIX VOLUME SET WHICH INCLUDES:

**VOLUME I: FIELD OPERATIONS (FO)
VOLUME II: GROUNDWATER (GW)
VOLUME III: GEOTECHNICAL (GT)
VOLUME IV: SURFACE WATER (SW)
VOLUME V: ECOLOGY (EE)
VOLUME VI: AIR (AP)**

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ADMIN RECORD

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By

Date

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[Signature]

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FIELD MEASUREMENTS OF SURFACE WATER FIELD PARAMETERS

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TITLE
FIELD MEASUREMENT OF SURFACE
WATER FIELD PARAMETERS

Approved By

J. B. Bernald
(Name of Approver)

MAY 12 1992

(Date)

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2.0 PURPOSE AND SCOPE

This standard operating procedure (SOP) describes procedures that will be used at the Rocky Flats Plant (RFP) to obtain measurements of surface water parameters in the field. These parameters are temperature, dissolved oxygen, pH, alkalinity, specific conductance, total residual chlorine, free chlorine, turbidity, hardness, and nitrates. This SOP describes field measurement procedures, personnel responsibilities and qualifications, and quality assurance/quality control (QA/QC).

3.0 RESPONSIBILITIES AND QUALIFICATIONS

Personnel measuring surface water field parameters will be geologists, hydrologists, engineers, or field technicians with an appropriate amount of applicable field experience or on-the-job training under supervision of another qualified person.

4.0 REFERENCES

4.1 SOURCE REFERENCES

The following is a list of references reviewed prior to the writing of this procedure.

A Compendium of Superfund Field Operations Methods EPA/540/P-87/001 U.S. Environmental Protection Agency Washington, D C 1987

Data Quality Objectives for Remedial Activities Development Process EPA/540/G-87/003. U.S. Environmental Protection Agency Washington, D C 1987

L.R. Kister and W.B. Garrett Field Guidelines for Collection, Treatment and Analysis of Water Samples, Arizona District U.S. Geological Survey, Water Resources Division. November 1984

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HACH DR/2000 Spectrophotometer Handbook, 1988

HACH ONE Electrode System Manual 9-8-89-SED HACH Company Loveland, CO. 1989

HACH Quick Reference Card K81-2ED HACH Company Loveland, CO

Instruction Manual Model 44600 Conductivity/TDS meter 5-23-89-6ED HACH Company
Loveland, CO 1989

Methods for Chemical Analysis for Water and Waste EPA-600/4-79-020 March 1983. Methods
150 1 and 330 5

Standard Methods for the Examination of Water and Wastewater 16th Edition. Method 212 1985

The Environmental Survey Manual DOE/EH-0053 Appendix E, "Field Protocols and Guidance"
U S Department of Energy Washington, D C August 1987

NPDES/FFCA Operations Sampling Plan Environmental Management Surface Water Division,
Rocky Flats (In Progress)

4.2 INTERNAL REFERENCES

Related SOPs cross-referenced by this SOP are as follows

- SOP FO.3, General Equipment Decontamination
- SOP FO 7, Handling of Decontamination Water and Wash Water

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- SOP FO 13, Containerizing, Preserving, Handling, and Shipping of Soil and Water Samples
- SOP SW.3, Surface Water Sampling

5.0 FIELD MEASUREMENT PROCEDURES

Calibration procedures for the equipment described in this section are found in Appendixes SW.2A and SW 2B

5.1 TEMPERATURE

Temperature measurements will be made with a high quality mercury-filled thermometer or thermistor having an analog or digital readout device. This thermometer is to have been standardized by comparison with a thermometer calibrated against a National Institute of Standards and Technology (NIST) calibrated thermometer. All temperature-measuring devices will be scaled to indicate degrees Celsius and marked as appropriate to meet data quality objectives. Glass thermometers will be transported in a protective case to prevent breakage. Thermometers will be Teflon® coated safety type, 305 mm in length, and scaled from -20°C to +110°C in 1° increments (VWR CAT No. 61017-823 or equivalent). Field thermometers will also be enclosed in an armored casing to prevent breakage (VWR CAT No. 61017-562 or equivalent).

Temperature measurements made for the purpose of providing adjustment factors for other field parameters will be conducted simultaneously with those related measurements. Volumes and methods of collection will be determined by the procedural requirements of the primary field measurement taken. Thermometer or thermistors used in the field will be standardized at least monthly against an NIST traceable thermometer.

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- Sampling personnel shall wear chemical-resistant gloves, which will be disposed of between sites, when performing field temperature measurements
- Only mercury-filled thermometers or thermistors that are in calibration will be used
- Inspect the thermometer before each field trip to ensure that there are neither cracks in the glass nor air spaces or bubbles in the mercury
- If a thermometer should be broken in the field, the location will be noted in the logbook and the site supervisor and health and safety officer will be notified immediately
- Allow the thermometer or thermistor enough time to equilibrate to outside temperature when removed from a field vehicle
- Insert the thermometer or thermistor into the stream. Swirl the thermometer or thermistor if the medium is calm and take the temperature reading when the mercury column or digital readout stabilizes
- Record the temperature reading in the field logbook to the nearest $\pm 0.5^{\circ}\text{C}$
- Decontaminate the thermometer or thermistor in accordance with SOP FO.3, General Equipment Decontamination

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- Liquids from decontamination operations will be handled in accordance with SOP No FO 7, Handling of Decontamination Water and Wash Water

5.2 TOTAL RESIDUAL CHLORINE (TRC)

Collect a representative 500-ml grab sample from the collection point into a sample bottle in accordance with SOP SW.3, Surface Water Sampling. Pour off approximately 50 ml to be used to determine TRC concentration. Cap remaining sample and retain in a cool location for later use in determining pH, alkalinity, and other field parameters.

Samples for analytes that are susceptible to changes in chemical composition due to high levels of TRC must be specially preserved when this condition exists. Before collection of samples at a sampling site, the TRC concentration must be measured. At sites where the concentration is measured to be ≥ 0.20 mg/l, volatile organic compounds (VOCs), cyanide and BNA samples will be preserved in accordance with SOP FO 13, Containerizing, Preserving, Handling, and Shipping of Soil and Water Samples. In the event that insufficient water is available to collect the entire sample suite, TRC should still be measured before sampling to determine sample preservation requirements. It is not necessary to measure TRC first if VOCs, cyanide or BNA are not being collected. If the TRC concentration exceeds 0.50 mg/l for any NPDES discharge points, contact the site supervisor immediately. The site supervisor will then contact the Environmental Management Surface Water Division at Rocky Flats.

TRC measurements at RFP will be conducted with a HACH DR/2000 spectrophotometer or equivalent. See equipment manufacturer's instructions for equipment-specific procedures. Take care to ensure that ampul tips are broken under water.

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5.3 TOTAL FREE CHLORINE (TFC)

The procedure for the measurement of this field parameter is included here to provide a reference for programs which may include this parameter (i.e, NPDES sampling)

TFC measurements at RFP shall be conducted with a HACH DR/2000 Spectrophotometer or equivalent See equipment manufacturer's instructions for equipment-specific procedures

5.4 DISSOLVED OXYGEN (D.O.)

5.4.1 YSI Model 57 D.O. Meter

Procedures for the YSI Model 57 D O meter will be as follows

- Inspect the membrane before each field use for air bubbles, oily film, and/or holes If the membrane is defective, it must be replaced and soaked before recalibration in accordance with manufacturer's literature
- Calibrate the meter at each site prior to use Instructions for the calibration procedure are included in Appendix SW.2B
- Perform an in-situ measurement by placing the D.O. electrode into the medium to be measured and reading the D O meter to the nearest 0.1 mg/l. Record the D O concentration and the range setting of the D O meter
- If the D.O. meter is equipped with an operational thermometer, read the water temperature at the time the D.O. is measured. If the meter does not provide a

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temperature reading, measure and record the water temperature as described in Subsection 5.1

- Consult Table 1 of Appendix SW.2B for the solubility of oxygen at the recorded temperature. If the measured D.O. value exceeds the solubility of oxygen at the given temperature, the D.O. value should be verified in the field by another method. The HACH DR/2000 can be used for this purpose. Consult the HACH manual for detailed instructions. Record the second D.O. value in the comment section of the field data collection form.
- Protect the YSI D.O. probe when not in use to prevent the membrane from drying out or freezing.
- Sampling tools, instruments, and equipment will be protected from sources of contamination before use and decontaminated after use as specified in SOP FO.3, General Equipment Decontamination.
- Sampling personnel shall wear chemical-resistant gloves, which will be disposed of between sites, when performing field D.O. measurements.
- Manufacturer's operating manuals and calibration procedures will be followed.

5.4.2 Hach DR/2000 Spectrophotometer

Dissolved oxygen may be measured using the Hach DR/2000 Spectrophotometer. See the manufacturer's instructions for specific procedures.

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5.5 pH

Measurements of pH are affected by changes in temperature. Mechanical and chemical changes in pH measuring electrodes due to temperature change can cause erroneous pH readings. The HACH One pH meter is a temperature compensating device. This or similar devices should be used for pH measurement to avoid introducing error.

The pH of water is also affected by exposure to the atmosphere. Carbon dioxide can escape from an exposed grab sample, thus altering pH in the sample. As described in Subsection 5.2, Total Residual Chlorine, the grab sample used for field parameter measurements should be capped and retained in a cool location to avoid pH changes.

pH is a measure of the activity of hydrogen ions in solution. Water samples will have varying ionic species and ionic strength, both of which affect the hydrogen ion activity. Accurate determination of pH is difficult in high ionic strength solutions due to this phenomenon. Proper electrode conditioning is necessary when measuring the pH of high ionic strength solutions.

Meters will be calibrated daily in the laboratory or field trailer prior to field use. Meters will also be checked against a pH 7.0 buffer in the field prior to use at each sampling site. Buffer solutions will be changed periodically for calibration checks.

If sample pH is less than 6 or greater than 9 pH units at NPDES discharge points, report results immediately to site supervisor, who will then report to the Environmental Management Surface Water Division at Rocky Flats.

Alkalinity measurements will accompany and immediately follow pH measurements detailed in this procedure and are to be considered an extension of the pH measuring process; not as an independent operation.

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Measurements in the field will be performed in the following manner (EPA method 150.1):

- Before each field activity, check the meter for cracked or fouled electrodes and battery condition in accordance with manufacturer's recommendations
- Check the instrument prior to use at the site by observing the reading obtained with a pH 7.0 buffer solution. The probe shall be free of contamination and dry before insertion into the buffer to limit cross-contamination and/or dilution of the solution. Recalibrate the instrument if pH is more than 2 pH units greater or less than buffer pH for the given temperature
- Thoroughly rinse the electrode and temperature probe with distilled water and remove excess water

Note When measuring samples of high ionic strength, condition the electrode after cleaning by dipping it into a portion of the sample water to be measured. After one minute, remove the electrode, blot dry, and then immerse in a fresh portion of the sample.

Sulfuric acid reagent (0.1600N or 1.600N) will be used in the alkalinity titrations. The 0.1600N H_2SO_4 reagent is preferable for alkalinities less than 100 mg/l. Above 100 mg/l, the 1.600N H_2SO_4 is preferable due to time constraints, but this higher concentration of acid is only to be used after previously recorded titrations indicate alkalinity greater than 100 mg/l.

- If the 0.1600N reagent has been selected, measure 50 ml of the 500-ml sample in a volumetric flask, then pour it into a 100-ml beaker containing a magnetic stir bar. This sample must be exactly 50 ml.

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- If the 1 600N reagent has been selected, place 50 ml of the grab sample into a 100-ml beaker as above, or place 100 ml into a 250-ml beaker containing a magnetic stir bar. Leave the magnetic stirring device off until the alkalinity titration procedure has begun. The sample, whether 50 ml or 100 ml, is measured with a volumetric flask and must be exact.
- Place the electrode and the temperature probe into a beaker containing 100 ml of the 500-ml sample and swirl the electrode at a constant rate until the meter reading stabilizes. The stirring rate should be maintained so as to minimize the surface disturbance of the sample.
- Note and record the indicated temperature of the sample to the nearest $\pm 0.1^{\circ}\text{C}$, and the pH to the nearest ± 0.1 pH unit.
- Rinse the electrode and temperature probe thoroughly with distilled water and store in accordance with manufacturer's recommendations.
- Sampling tools, instruments, and equipment will be protected from sources of contamination before use and decontaminated after use as specified in SOP FO.3, General Equipment Decontamination.
- Sampling personnel shall wear chemical-resistant gloves, which will be disposed of between sites, when performing field pH measurements.

5.6 ALKALINITY

Alkalinity measurements will accompany and immediately follow pH measurements detailed in Subsection 5.5 of this procedure. Titration with an appropriate acid reagent will be used to measure

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alkalinity Alkalinity measurements at the RFP will be conducted with a HACH digital titrator or equivalent. Follow the general procedures described below and see equipment manufacturer's instructions for detailed equipment-specific titration procedures.

- Sampling personnel will wear chemical-resistant gloves, which will be disposed of between sites, when performing alkalinity measurements.
- Determine the appropriate end-point pH according to alkalinity species in question (4.5 or 4.5 and 8.3 if initial pH > 8.3).
- Prepare sample and titration assembly.
- Titrate to endpoint pH by making smaller additions of acid as the endpoint is approached.
- Use appropriate conversions based on reagent normality and sample size to calculate alkalinity.

5.7 SPECIFIC CONDUCTANCE (SC)

Conductance is a measure of the ability of an aqueous solution to conduct electrical current and is expressed in reciprocal ohms (mhos). The International System of Units uses the siemen(s) to represent mhos. The siemen will be the unit used in this SOP.

The physical dimensions of a conductance measuring probe define the cell constant for the probe. When this constant is known and applied, conductance is converted to units of specific conductance (SC) (a.k.a., conductivity). Most waters have a SC much less than 1 siemen, therefore, data will be reported in millisiemens (mS)/cm or microsiemens (μ S)/cm.

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The Hach 44600 Conductivity/TDS meter has been pre-set in the factory with a cell constant of 1.0. The cell constant is determined by using a potassium chloride (KCl) standard solution. The temperature coefficient (used in cell constant determination) of most waters is only approximately the same as that of the KCl solution. The greater the temperature difference between the KCl standard and the sample, the greater the uncertainty in the measurement of SC. For greatest measurement accuracy, the KCl solution and the sample should both be 25 °C.

SC increases with temperature. The meter is temperature compensating, correcting readings to the standard temperature 25°C, over the temperature range 0-100°C. Thus, no error should be introduced in conductivity measurements due to temperature.

The HACH 44600 Conductivity/TDS meter will be standardized each day before field activities against a 1000 $\mu\text{S}/\text{cm}$ solution and a 10,000 $\mu\text{S}/\text{cm}$ solution. These standards define the limits, between which conductivity readings are reliable. Solutions of particularly high ionic strength (with conductivity > 10,000 $\mu\text{S}/\text{cm}$) should be diluted to 50 percent strength until readings fall within the prescribed limits.

The following method will be used to measure SC in the field (EPA method 330.5)

- Before each field activity, check the meter for damage to the probe and for weak batteries in accordance with manufacturer's recommendations.
- Calibrate the meter in the field prior to use at each site. Instructions for the calibration procedure are included in Appendix SW.2A.
- Thoroughly rinse the probe in distilled water and remove excess water by gently shaking and drying with clean paper towel before immersion in the sample.

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- A 250-ml portion of the 500-ml grab sample described in Subsection 5.2, Total Residual Chlorine, will be measured for SC
- Immerse the probe into the sample to a depth of at least 1 inch below the surface of the sample. Agitate the probe gently to dislodge any trapped air bubbles and allow the meter reading to stabilize
- Record the temperature and the temperature-compensated reading in the daily field logbook
- Rinse the probe thoroughly with distilled water after use and dry with a clean paper towel
- Sampling tools, instruments, and equipment will be protected from sources of contamination before use and decontaminated after use as specified in SOP FO.3, General Equipment Decontamination
- Sampling personnel shall wear chemical-resistant gloves, which will be disposed of between sites, when performing field conductivity measurements

5.8 SILICA (SiO_2)

In the event that SiO_2 measurements are needed, the Silicomolybdate method is used to measure silica in the range of 0 - 100 mg/l, and the Heteropoly Blue method measures silica in the 0 - 16 mg/l range. Silica measurements at RFP will be conducted with a HACH DR/2000 spectrophotometer or equivalent. See equipment manufacturer's instructions for equipment-specific procedures.

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5.9 TURBIDITY

This procedure describes the measurement of turbidity using the HACH DR/2000 Spectrophotometer absorptometric method. The turbidity test measures an optical property of the water sample which results from the scattering and absorbing of light by the particulate matter present. The amount of turbidity registered is dependent on such variables as the size, shape, and refractive properties of the particles. This procedure is calibrated using formazin turbidity standards, and the readings are in terms of formazin turbidity units (FTU).

- Sampling personnel will wear chemical-resistant gloves, which will be disposed between sites, when performing turbidity measurements.
- Enter the stored program number for turbidity; press "750 Read/Enter". The display will show "Dial nm to 450".
- Rotate the wave length dial until the small display shows "450 nm".
- Press "Read/Enter". The display will show "FTU Turbidity".
- Pour 25 ml of deionized water (blank) into a sample cell.
- Place the blank into the cell holder and close the light shield.
- Press "zero" and the display will show "wait" and then "0. FTU Turbidity".
- Agitate the sample designated for turbidity measurement so that all sediments are suspended. Pour 25 ml of the sample into another clean sample cell; place into the cell holder and close the light shield.

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- Press "Read/Enter" and the display will show "wait" and then the result in FTUs will be displayed
- Read value and record in logbook

5.10 HARDNESS

Hardness is defined as a characteristic of water which represents the total concentration of calcium and magnesium ions expressed as their calcium carbonate equivalent (mg/l CaCO_3). Calcium and magnesium ions are the principle causes of water hardness. Although less common in natural waters than calcium and magnesium, other ions (i.e., iron, aluminum, manganese, strontium, zinc and hydrogen) are capable of producing the same hardness effect and will be included in the results.

Ethylenediaminetetraacetic acid and its sodium salts (abbreviated EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of a dye such as Erichrome Black T or Calmagite is added to an aqueous solution containing calcium and magnesium ions at a pH of 10.0 ± 0.1 , the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the magnesium and calcium has been complexed the solution turns from wine red to blue, marking the end point of the titration. Magnesium ions must be present to yield a satisfactory end point. To insure this, a small amount of complexometrically neutral magnesium salt of EDTA (Mg-CDTA) is added to the buffer (HACH Buffer Solution, Hardness 1), this automatically introduces sufficient magnesium.

This procedure describes the measurement of hardness using the digital titration method for an expected concentration range of 0-250 mg/l total hardness.

- Sampling personnel shall wear chemical-resistant gloves, which will be disposed between sites, when performing hardness measurements.

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- Attach a clean 90° delivery tube to a 0.800M EDTA Titration Cartridge. Twist the cartridge onto the titrator body. Hold the Digital Titrator with the tip pointing up. Flush out the air in the delivery tube by turning the delivery knob until a few drops of titrant are ejected from the tube and no visible air remains in the cartridge. Reset the counter to zero and wipe the tip. Attach the digital titrator to the laboratory stand.
- Using a graduated cylinder, add 100 ml of sample into a 125-ml erlenmeyer flask containing a magnetic stir bar. Turn on the magnetic stirrer and maintain a rate so as to minimize the surface disturbance of the sample.
- Add 2 ml of Hardness 1 Buffer Solution and swirl to mix. The addition of this buffer will bring the pH of the solution to 10.0 ± 0.1 .
- Add the contents of one ManVer 2 Hardness Indicator Powder Pillow and swirl to mix. The color of the solution should turn to wine red.
- Place the delivery tube tip into the solution and titrate the sample until the color changes from red to blue. Titrate slowly toward the end point as it takes time for the reaction and color change to take place, especially in cold water. A limit of 5 minutes is set for the duration of the titration to minimize the tendency toward CaCO_3 precipitation.
- Read the concentration of total hardness (as mg/l CaCO_3) from the digital counter and record the results.

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5.11 NITRATES

Nitrate represents the most completely oxidized state of nitrogen commonly found in water. Nitrate forming bacteria convert nitrites (NO_2) into nitrates (NO_3) under aerobic conditions and lightning converts large amounts of atmospheric nitrogen (N_2) directly to nitrates. High levels of nitrate in water indicates biological wastes in the final stages of stabilization or run-off from heavily fertilized fields. Nitrate rich effluents discharged into receiving waters can degrade water quality by encouraging excessive growth of algae.

This procedure describes the measurement of Nitrate using the HACH DR/2000 Spectrophotometer absorption method.

- Sampling personnel shall wear chemical-resistant gloves, which will be disposed between sites, when performing Nitrate measurements.
- Enter the stored program number for Nitrate, High Range, press "360 Read/Enter". The display will show "Dial nm to 500".
- Rotate the wave length dial until the small display shows "500 nm".
- Press "Read/Enter". The display will show "mg/l N NO_3 - H AV".
- Collect at least 40 ml of sample in a 50 ml beaker. Fill the rubber ampul cap with sample. Fill a NitraVer 5 Nitrate AccuVac Ampul with sample by immersing it in the beaker and breaking the tip along the side of the beaker. Keep the tip immersed while the ampul fills completely. Cover the ampul with the cap.

FIELD MEASUREMENTS OF SURFACE WATER FIELD PARAMETERS

EG&G ROCKY FLATS PLANT
EMD MANUAL OPERATION SOP

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- Press "Shift Timer" A one minute mixing period will begin. Invert the ampul repeatedly until the timer beeps Wipe off any liquid or fingerprints
- When the timer beeps, press "Shift Timer" A five minute reaction time will begin An amber color will develop if nitrate-nitrogen is present
- Fill a zeroing vial with at least 10 ml of sample (the blank)
- When the timer beeps, the display will show "mg/l N NO₃ - H AV" Place the blank into the cell holder and close the light shield
- Press "Zero" and the display will show "Wait" and then "0 0 mg/l N NO₃ - H AV"
- Place the AccuVac ampul into the cell holder and close the light shield
- Press "Read/Enter" and the display will show "Wait", then the nitrate result in mg/l nitrate-nitrogen (NO₃-N) will be displayed Record the value

6.0 QUALITY ASSURANCE/QUALITY CONTROL

Quality assurance (QA) and quality control (QC) activities will be accomplished according to applicable project plans as well as quality requirements presented in this SOP. Equipment inspection and calibration QC requirements for each field parameter measurement procedure are described in Section 5.0 of this SOP

FIELD MEASUREMENTS OF SURFACE WATER FIELD PARAMETERS

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7.0 DOCUMENTATION

Information required by this SOP will be documented on the Surface Water Data Collection Field Notes Form (Form SW 1A) or the NPDES daily log sheets. NPDES log sheets are described in the NPDES/FFCA Operations Sampling Plan. Use of these forms is described in SOP SW 1, Surface Water Data Collection Activities. Data required by this SOP include calibration records and measurements of temperature, D O, pH, alkalinity, SC, TRC, TFC, turbidity, hardness and nitrates.

APPENDIX SW.2A

**CALIBRATION AND STANDARDIZATION
PROCEDURES**

APPENDIX SW.2A - CALIBRATION AND STANDARDIZATION PROCEDURES

These instructions are for the use of persons who will use these field measurements on a daily basis and are not intended to either supersede or supplement required periodic laboratory calibration or manufacturer's guidance for initiation of new equipment. In all cases, care will be taken to ensure that reagents and standards are employed that have not exceeded their expiration dates.

Standardization is the process of determining the deviation between the known value of a standard and the value for that standard measured by an instrument. Calibration is the process of adjusting an instrument by that deviation such that known and measured values for a standard are equivalent.

A.1 THERMOMETER AND THERMISTOR STANDARDIZATION

Measured values of specific conductance, pH, and D O are all temperature-dependent and the instruments used to measure these parameters also measure and/or compensate for temperature effects. Therefore, accuracy of the temperature measurements obtained with these instruments, as well as any thermometers utilized to measure temperature, must be determined and a record maintained.

Laboratory temperature standardization of these instruments will be performed and documented on each instrument monthly. The thermometers and thermistors in each instrument being utilized will be standardized against a thermometer traceable to an NIST calibrated thermometer. Accuracy will be determined throughout the expected working range (generally 0° to 35°C). A three-point standardization within the working range will be used to verify accuracy. The following procedure will be followed:

- 1 Have ready solutions of water in the following temperature ranges. 0 to 10 °C, 15 to 25 °C, 30 to 40 °C
- 2 Immerse the thermistor or thermometer and the NIST traceable thermometer into the 0-10 degree C solution. After allowing time for readings to stabilize, record the readings of the NIST traceable thermometer and the thermistor or thermometer being standardized.
- 3 Repeat Step 2 using the 30-40 degree C solution. If the temperature value of the thermistor can be adjusted from external control knobs on the instrument, then

the temperature reading of the instrument should be adjusted to the temperature reading of the NIST traceable thermometer and the adjusted value noted in the logbook

- 4 Repeat Step 2 using the 15-25 degree C solution.
- 5 Thermometers must read within $\pm 1^{\circ}\text{C}$ of the NIST traceable thermometer
Thermometers reading outside this range will not be used for field measurements
Thermistor readings should be within $\pm 2^{\circ}\text{C}$ of the NIST traceable thermometer
Instruments with temperature readings outside this range should be returned to the factory for calibration

A.2 CALIBRATION OF THE HACH DR/2000 SPECTROPHOTOMETER

The HACH DR/2000 Spectrophotometer, used in the measurement of total residual chlorine and other parameters, is pre-calibrated by the manufacturer. Consult the HACH instrument manual in the event instrument problems occur.

A.3 CALIBRATION OF THE YSI DISSOLVED OXYGEN (D.O.) METER

A.3.1 Calibration in the Base Laboratory

Calibration of the D O meter will be performed daily before leaving the base laboratory to check the meter to see if it is in proper working order. This calibration procedure is as follows:

- Prepare an oxygen-saturated calibration solution by agitating 200 ml to one liter of distilled water vigorously for approximately five minutes or agitate in blender for 30 seconds.
- Turn the instrument to the "Red Line" setting and adjust the meter needle to correspond to the red line on the instrument.
- Turn the instrument to the zero setting and adjust the meter reading to read zero.
- Place the probe into the saturated water. Read both the temperature and the D O value. Consult the chart in the manufacturer's literature and adjust the

meter scale to the value of saturation at the measured temperature and atmospheric pressure

- If the instrument cannot be calibrated to within ± 10 percent of the standard value, it will require maintenance and recalibration prior to use

A.3.2 Field Calibration

Calibration of the D O. meter in the field will be performed at each sample site with an Air Calibration Chamber. This device (YSI number 5075, or equivalent) permits calibration of the D O meter at the temperature of the water in which the D O is to be measured, thereby minimizing errors due to temperature differences. Steps to follow for calibration are listed in the instruction manual for the YSI Model 57 Dissolved Oxygen Meter. Please refer to page 12 of Appendix SW.2B for calibration procedures. Figure 8, also on page 12, is a useful diagram found in Appendix SW.2B, illustrating the parts of the calibration unit and the D O. probe, and is referred to in the calibration text.

A.4 CALIBRATION OF THE HACH ONE pH METER

All pH instruments will receive a daily pre-use calibration and a post-use standardization, both of which will be recorded in field logbooks and base lab notebooks. Before use at each site, the instrument will be checked against the pH 7.0 standard buffer. If the instrument reading is not within ± 0.2 pH units of the standard buffer, the instrument must be recalibrated.

These procedures will require the use of buffer solutions of the type listed below as required by the manufacturer.

- pH 4 buffer - potassium hydrogen phthalate
- pH 7 buffer - potassium phosphate, monobasic, and sodium phosphate, dibasic
- pH 10 buffer - sodium carbonate-bicarbonate

Instructions for pre-use calibration are

- Turn meter on by pressing POWER key. The display will light.
- Depress the pH key.

- Next, press the AUTO/MANUAL key. The AUTO indicator should now be lit, the S1 and pH indicators flashing, and all zeroes appearing on the display.
- Remove the cap from the electrode/temperature probe and place the probe into a stirring pH 4 buffer solution and press the potassium chloride dispenser button. Allow approximately 30 seconds after dispensing electrolyte before performing the next operation.
- The STANDARD key may now be depressed. Wait until the pH indicator stops flashing. The S2 indicator will begin flashing and the actual pH value will appear on the display. (Note: The display will indicate the actual pH value at the actual temperature of the buffer solution. Consult the variation chart provided with the instrument to determine if the instrument is within appropriate parameters.) Record the indicated pH value and temperature, and the adjusted pH value of the buffer at the indicated temperature from the chart in the logbook.
- Rinse the electrode/temperature probe with distilled water and blot dry with a clean paper towel.
- Place the electrode/temperature probe into a stirring pH 10 buffer solution and again press the dispenser button. Wait at least 30 seconds before continuing.
- Once again, press the STANDARD key. S2 will stop flashing. After the pH indicator stops flashing, the actual pH value at the given temperature will appear on the display. Record the indicated pH value and temperature, and the adjusted pH value of the buffer in the logbook.
- If, during calibration, the digital readout fails to stabilize in any of the solutions, replacement of the pH electrode and/or the temperature probe may be necessary.
- DO NOT PRESS ANY KEY OTHER THAN THE pH KEY at this point or the entire calibration procedure will be nullified.
- Press the pH key.

- Rinse the electrode/temperature probe with distilled water and blot dry with a clean paper towel
- Place the cap on the probe and place the instrument in the case
- This instrument is now ready to perform pH measurements

At the end of the day, a post-use standardization will be performed on the instrument. The pH of the 4, 7, and 10 buffer standards will be measured with the instrument. The values of the standards and measured values will be recorded in the appropriate base lab logbook. Measurement and documentation of the standard buffers will ensure and document that the instrument is still functioning within acceptable limits. Acceptable limits for the instrument will be within ± 0.2 pH units of the standard. If the instrument does not consistently stay within these acceptance limits, then it will become necessary to calibrate the instrument prior to use at every site. The sample manager will consult with the site supervisor and QA officer, and the QA officer will decide if calibration at each site is necessary.

A.5 STANDARDIZATION INSTRUCTIONS FOR THE HACH DIGITAL TITRATOR AND ALKALINITY TITRANT REAGENT USED FOR THE FIELD MEASUREMENT OF ALKALINITY

Calibration of the pH meter will first be accomplished in accordance with Section A.4 of this Appendix. Standardization of the digital titrator with sulfuric acid reagent (0.1600N or 1.600N) will be done monthly or each time a new lot of reagent is received. The standardization procedure will be accomplished as follows:

- Measure 50 ml of a 100-ppm alkalinity standard in a 50 ml volumetric flask.
- Transfer this solution into a clean beaker containing a magnetic stir bar.
- Place the beaker on a magnetic stir plate.

-
- This procedure assumes that the concentration of the purchased alkalinity standard is correct, and therefore the reagent titrant is the solution being standardized.

- Load the digital titrator with the appropriate reagent cartridge (0.1600N or 1.600N H_2SO_4)
- Position the pH electrode and digital titrator-dispensing tube into the beaker containing the 50 mls of the 100-ppm alkalinity standard.
- Adjust digital count setting knob to ensure that the counts on the digital titrator are set at zero
- Turn on the stir plate and stir the 50 mls of the 100-ppm alkalinity standard at a rate that will keep the solution well mixed but not cause splashing.
- Measure the initial pH of the solution
- Slowly add titrant to the solution. Decrease the rate of addition as the pH approaches 5.0
- Continue to add titrant until the pH lowers to 4.5
- Record the amount of titrant (number of digital counts) required to bring the pH to 4.5
- Multiply the number of digital counts by the appropriate conversion factor (0.2 if the 0.1600N H_2SO_4 was used, and 2.0 if the 1.600N reagent was used)
- Record this result as ppm alkalinity in the base lab notebook.
- If the value obtained is not within ± 10 percent of the alkalinity standard, then either the acid reagent concentration is not correct or the digital titrator is not dispensing accurately. The problem must be resolved and corrected. Acceptable titration to within ± 10 percent of the alkalinity standard will be obtained before the titrator and sulfuric acid reagent is to be used.

A.6 CALIBRATION INSTRUCTIONS FOR HACH MODEL 44600 CONDUCTIVITY/TDS METER

The HACH Model 44600 Conductivity/TDS meter will be calibrated daily in the laboratory prior to use in the field. The instrument will not receive a post-standardization since it will be standardized in the field prior to use at each site.

Note A reading within ± 10 percent of the value of the conductivity standard (10,000 or 1,000 $\mu\text{S}/\text{cm}$) and 100 $\mu\text{S}/\text{cm}$ for the zero conductivity standard (distilled water) is within acceptable limits and the meter may be used for field measurements. However, the meter should always be set to read as close as possible to the concentration of the standard. The procedure for calibration of the meter is as follows:

- Press the POWER 1 key and CND key. Verify that the LO BAT indication does not appear. If LO BAT is indicated, the battery will be replaced.
- Place the instrument probe into the calibration solution to a depth of 1 inch or more beyond the vent holes and agitate vertically to release entrapped air bubbles.
- Adjust the displayed reading to the value of the conductivity standard and allow the reading to stabilize.
- Record the display reading in the logbook.
- Press the °C key and record the display reading in the logbook.
- Remove the probe from the solution and rinse thoroughly with distilled water.
- Place the probe into fresh, distilled water and repeat the steps indicated above, recording the appropriate readings. This establishes a zero point or lower bracket reading.
- The instrument is now ready to perform measurements.
- Repackage the instrument and probe in the case for transport.

Note: In the field the instrument will be standardized against an appropriate 10,000 or 1,000 $\mu\text{S}/\text{cm}$ standard solution

A.7 STANDARDIZATION INSTRUCTIONS FOR THE HACH DIGITAL TITRATOR AND HARDNESS TITRANT REAGENT USED FOR THE FIELD MEASUREMENT OF HARDNESS*

Standardization of the digital titrator with EDTA reagent (0.800 M) shall be done monthly or each time a new lot of reagent is received. The standardization procedure will be accomplished as follows

- Attach a clean 90° delivery tube to a 0.800M EDTA Titration Cartridge. Twist the cartridge onto the titrator body. Hold the Digital Titrator with the tip pointing up. Flush out the air in the delivery tube by turning the delivery knob until a few drops of titrant are ejected from the tube and no visible air remains in the cartridge. Reset the counter to zero and wipe the tip. Attach the digital titrator to the laboratory stand.
- Using a graduated cylinder measure 20 ml of the Calcium Standard Solution, 1000 mg/l as CaCO_3 .
- Transfer this solution into a clean 50 ml beaker containing a magnetic stir bar.
- Turn on the magnetic stirrer and maintain a rate so as to minimize the surface disturbance of the sample.
- Add 2 ml of Hardness 1 Buffer Solution and swirl to mix.
- Add the contents of one ManVer 2 Hardness Indicator Powder Pillow and swirl to mix. The color of the solution should turn to wine red.
- Place the delivery tube tip into the solution and titrate the sample until the color changes from red to blue. Titrate slowly toward the end point as it takes time for the reaction and color change to take place, especially in cold water. A limit of 5 minutes is set for the duration of the titration to minimize the tendency toward CaCO_3 precipitation.

- Read the concentration of total hardness (as mg/l CaCO_3) from the digital counter
- Record this result in the base lab notebook
- If the value is not within ± 10 percent of the hardness standard, then either the reagent concentration is not correct or the digital titrator is not dispensing accurately. The problem must be resolved and corrected. Acceptable titration to within ± 10 percent of the hardness standard will be obtained before the titrator and EDTA reagent is to be used

APPENDIX SW.2B

**INSTRUCTION MANUAL FOR YSI MODEL 57
DISSOLVED OXYGEN METER**

INSTRUCTION MANUAL
YSI MODEL 57
DISSOLVED OXYGEN METER



YSI Incorporated
Yellow Springs Instrument Co., Inc., Yellow Springs, Ohio 45387 USA
Phone 513 767-7241 / 800 343-HELP / Fax 513 767-9353 / Telex 205437

SUMMARY OF OPERATING INSTRUCTIONS

1 CALIBRATION

- A Switch instrument to OFF and adjust meter mechanical zero
- B Switch to RED LINE and adjust
- C Prepare probe for operation. Plug into instrument wait up to 15 minutes for probe to stabilize. Probe can be located in calibration chamber (see instruction manual) or ambient air
- D Switch to ZERO and adjust
- E Adjust SALINITY knob to FRESH
- F Switch to TEMP and read
- G Use probe temperature and true local atmospheric pressure (or feet above sea level) to determine correct calibration values from Table I and II (See pages 13 and 14)

EXAMPLE Probe temperature = 21°C. Altitude = 1000 feet From Table I the calibration value for 21°C is 8.9 mg/l From Table II the altitude factor for 1000 feet is approximately 96 The correct calibration value is

$$8.9 \text{ mg/l} \times 96 \text{ factor} = 8.54 \text{ mg/l}$$

- H Switch to desired dissolved oxygen range 0-5 0-10 or 0-20 and with calibrate control adjust meter to correct calibration value determined in Step G

NOTE It is desirable to calibrate probe in a high humidity environment See instruction manual for more detail on calibration and other instrument and probe characteristics

2 MEASUREMENT

- A Adjust the SALINITY knob to the salinity of the sample
- B Place the probe and stirrer in the sample and switch the STIRRER control to ON
- C When the meter has stabilized switch to the appropriate range and read DO
- D We recommend the instrument be left on between measurements to avoid necessity for repolarizing the probe

3. GENERAL CARE

- A Replace the instrument batteries when unable to adjust to red line Use (2) Eveready No 935 "C" size or equivalent
- B In the BATT CHECK position the voltage of the stirrer batteries is displayed on the red 0-10 scale Do not discharge below 6.0 Volts Recharge for 14-16 hrs with YSI No 5728 charger
- C Membrane will last indefinitely, depending on usage Average replacement is 2-4 weeks. Probe should be stored in humid environment to prevent drying out.
- D Calibrate daily

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GENERAL DESCRIPTION

The YSI Model 57 Dissolved Oxygen Meter is intended for dissolved oxygen and temperature measurement in water and wastewater applications but is also suitable for use in certain other liquids. Dissolved Oxygen is indicated in mg/l (milligrams per liter) on 0-5, 0-10 and 0-20 mg/l scales. Temperature is indicated in °C on a -5° to +45°C scale. The dissolved oxygen ranges are automatically temperature compensated for solubility of oxygen in water and permeability of the probe membrane, and manually salinity compensated.

The probes use Clark-type membrane covered polarographic sensors with built in thermistors for temperature measurement and compensation. A thin permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and certain other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

SPECIFICATIONS

I Instrument

Oxygen Measurement

Ranges 0-5, 0-10 and 0-20 mg/l (0-2, 5, 0-5 and 0-10 mg/l with YSI 5776 High Sensitivity Membrane)

Accuracy ±1% of full scale at calibration temperature (±0.1 mg/l on 0-10 scale), or 0.1 mg/l (whichever is larger)

Readability 0.25 mg/l on 0-5 scale, 0.5 mg/l on 0-10 scale, 0.1 mg/l on 0-20 scale

Temperature Measurement

Range -5° to +45°C

Accuracy ±0.5°C plus probe which is ±0.1°C

Readability 0.25°C

Temperature Compensation

±1% of D O reading for measurements made within ±5°C of calibration temperature

±3% of D O reading over entire range of -5 to +45°C probe temperature

System Response Time

Typical response for temperature and D O readings is 90% in 10 seconds at a constant temperature of 30°C with YSI 5775 Membranes. D O response at low temperature and low D O is typically 90% in 30 seconds. YSI 5776 High Sensitivity Membranes can be used to improve response at low temperature and low D O concentrations. If response time under any operating conditions exceeds two minutes, probe service is indicated.

Operating Temperature Range

Instrument and probe operating range is -5° to +45°C. Large ambient temperature changes will result in 2% loss of accuracy unless Red Line and Zero are reset.

Recorder Output

0 to 114, 136 mV. Recorder should have 50,000 ohms minimum input impedance.

Power Supply

The YSI Model 57 is powered by two disposable C size carbon zinc batteries (Eveready 935C or equal) providing approximately 1000 hour operation.

II Probe

Cathode Gold

Anode Silver

Membrane 001 FEP Teflon (0005 FEP Teflon available)

Electrolyte Half saturated KCl

Temperature Compensation (See SPECIFICATIONS I Instrument)

Pressure Compensation Effective 1/2% of reading with pressures to 100 psi (230 ft sea water)

Polarizing Voltage 0.8 volts nominal

Probe Current Air at 30°C = 19 microamps nominal

Nitrogen at 30°C = 15 microamps or less

III Accessories and Replacement Parts

YSI 5720A — Self Stirring BOD Bottle Probe

YSI 5750 — Non Stirring BOD Bottle Probe

YSI 5739 — Oxygen Temperature Probe for field use. Combine with one of the following 4 cables for desired lead length.

Detachable leads for use with YSI 5739

YSI 5740-10 10 Cable

YSI 5740-25 25 Cable

YSI 5740-50 50 Cable

YSI 5740-100 100 Cable

YSI 5740-150 150 Cable

YSI 5740-200 200 Cable

YSI 5721 — Battery and charger pack operates YSI 5791A and 5795A Submersible Stirrers

YSI 5791A — Submersible Stirrer for field use

YSI 5795A — Submersible Stirrer

YSI 5075A — Calibration Chamber for use with field probe,

YSI 5890 — Carrying Case

YSI 5775 — Membrane and KCl Kit Standard — includes 2 each 15 membrane packets (001 thick standard membranes) and a 30 ml bottle KCl with Kodak photo flo

YSI 5776 — Membrane and KCl Kit High Sensitivity — includes 2 each 15-membrane packets (0005 thick membranes) and a 30 ml bottle KCl with Kodak photo flo

YSI 5680 — Probe Reconditioning Kit

YSI 5945 — "O" Ring Pack — includes (6) "O" rings for each YSI DO Probe
YSI 5486 — Beater Boot Kit — includes (1) A-05486 Boot (1) A-05484
Tip (2) A-05485 Spring Used only on 5720A and discontinued 5420A

YSI 5986 — Diaphragm Kit for use only with YSI 5739 D O Probe
YSI 5735 — Adaptor makes it possible to use YSI 5739, YSI 5720A and
YSI 5750 Probes with discontinued YSI Models 51A, 54RC
and 548P

OXYGEN PROBES AND EQUIPMENT

There are three oxygen probes for use with the YSI Model 57 Dissolved
Oxygen Meters. Descriptions of where they are used are contained in the follow-
ing paragraphs

1 YSI 5739 D O Probe

The YSI 5739 probe is designed for use with the 5740 detachable cable and
replaces the discontinued YSI 5418, 5419, 5718 and 5719 probes (See Figure
1)

For user convenience the probe is equipped with a disconnecting cable to
facilitate changing cable lengths and replacing damaged cables or probes. The
probe and cable assembly is held together with a threaded retaining nut. The
connection is *not* designed for casual disconnection and should only be dis-
connected when necessary.

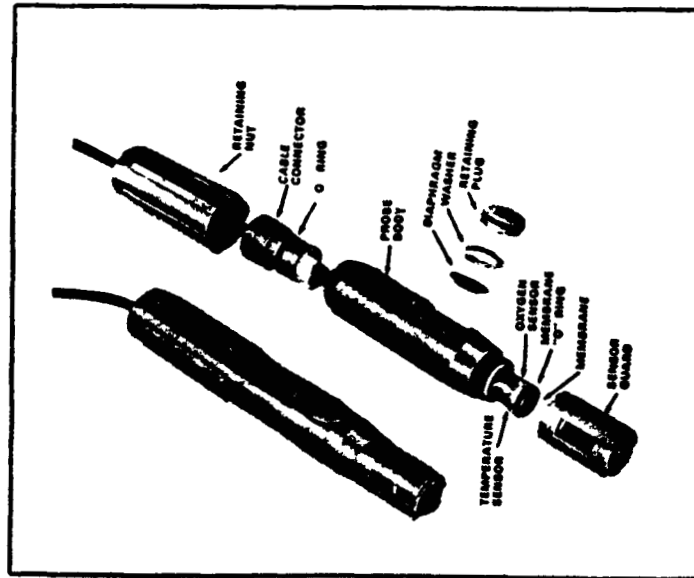


FIGURE 1

disconnect the cable unscrew the retaining nut and slide it down the cable
to expose the connector. Pull gently on the cable and connector until the connec-
tor comes away from the probe body.

To reassemble inspect the connector and "O" ring for cleanliness. If the "O"
ring is frayed or damaged remove it by squeezing it in the groove causing it to
bulge then roll it out of the groove and off the connector. A replacement "O" ring
is supplied with the cable.

Push the connector into the probe body, rotating it until the two halves mate.
A light coating of vaseline or silicone grease on the "O" ring will make
reassembly easier. Air trapped between the connector halves which may cause
them to spring apart slightly, is normal. Screw on the retaining nut *hand tight*
only. NOTE: If erratic readings are experienced disconnect the cable and inspect
for water. If present dry out and reconnect, replacing the "O" ring if necessary.

Pressure Compensation

The vent on the side of the probe is part of a unique pressure compensating
system that helps assure accurate readings at great depths of water. Pressure
compensation is effective to 1/2% of reading with pressures to 100 psi (230 ft
water). The quantity of air bubbles trapped under the membrane determines how
serious the pressure error will be, which is why proper preparation of the probe is
essential (See OPERATING PROCEDURES). The system is designed to accom-
modate a small amount of trapped air and still function properly, but the amount
should be kept to a minimum.

The compensating system normally does not require servicing and should not
be taken apart. However, if electrolyte is leaking through the diaphragm or if
there is an obvious puncture, the diaphragm must be replaced. A spare is
supplied with the probe. Using a coin unscrew the retaining plug and remove the
washer and the diaphragm, flush any salt crystals from the reservoir, install the
new diaphragm (convolution side in), replace the washer, and screw in the retain-
ing plug.

II. YSI 5720A B O D Bottle Probe

The YSI 5720A B O D Bottle Probe replaces the discontinued YSI 5420A
B O D Bottle Probe for measuring dissolved oxygen and temperature in standard
B O D bottles. It is provided with an agitator for stirring the sample solution,
available in models for 117VAC (95-135VAC, 50-60 Hz) or 230VAC (190-
250VAC, 50-60 Hz) operation (See Figure 2).

When using the probe, plug the agitator power supply into line power and the
probe plug into the instrument. With the agitator turned off place the tapered
probe end into the B O D bottle and switch agitator "ON" with switch on top of
probe. The probe should be operated with a *minimum* of trapped air in the B O D
bottle. A slight amount of air in the unstirred region at the top of the bottle may
be neglected but no bubbles should be around the thermistor or oxygen sensor.

Stirrer Boot

The probe uses a flexible stirring boot to transmit motion from the sealed
motor housing to the sample. If the boot shows signs of cracking or other
damage likely to allow leaking into the motor housing, the boot must be
replaced.

In fresh water applications boot life is normally several years, but this may be
shortened by exposure to hydrocarbons, moderate to strong acids or bases.

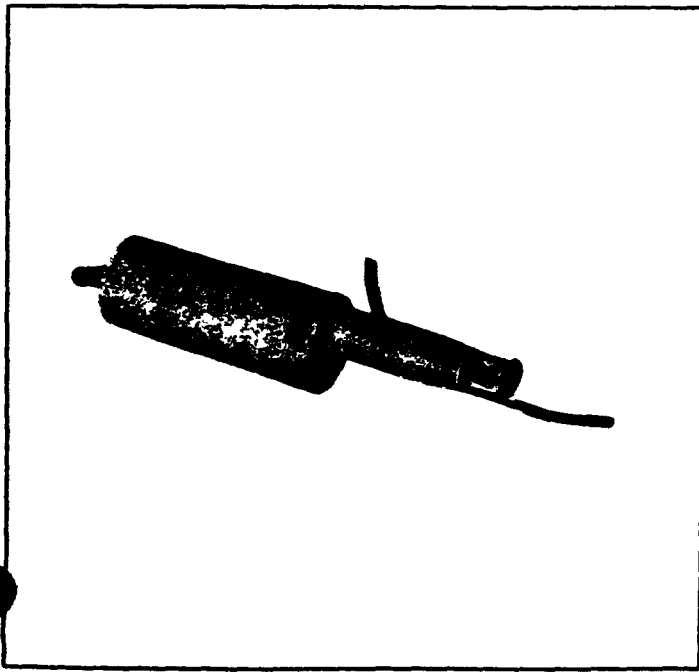


FIGURE 2

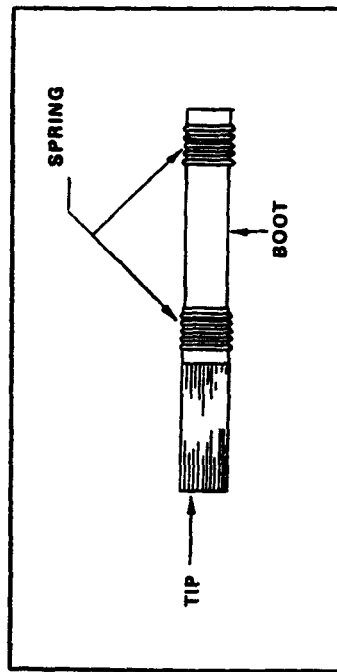


FIGURE 3

ozone or direct sunlight. For maximum life, rinse the boot after use in contaminated samples. (See Figure 3)

Boot replacement is as follows:

1. Pull off old assembly and clean shaft.
2. Slide on new assembly, making sure the back spring is on the grooved area of the shaft. A small amount of rubber cement may be used.
3. Check that there is sufficient clearance between the tip and the end of the shaft to permit turning without binding.



FIGURE 4

III YSI 5750 BOD Bottle Probe

The YSI 5750 BOD Bottle Probe replaces the discontinued YSI 5450 BOD Bottle Probe. It is similar to the YSI 5720A BOD Bottle Probe except that it does not have a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer. (See Figure 4)

IV Cable Adaptors

All YSI 5700 Series Probes are designed for direct use with the YSI Model 57 Dissolved Oxygen Meter.

V YSI 5791A and 5795A Submersible Stirrers

The YSI submersible stirrers are accessories that perform the function of stirring the sample being studied when making dissolved oxygen measurements in the field. The YSI 5791A stirrer can be used with the following dissolved oxygen probes: YSI 5418, 5419, 5718, 5719, and 5739. The YSI 5795A stirrer is only for use with the YSI 5739 Probe. (See Figure 5)

When a stirrer and probe are assembled, the stirrer agitates the sample directly in front of the sensor by means of a rotating eccentric weight which causes the spring-mounted hermetically sealed motor housing to vibrate. An impeller on the end of the motor housing flushes the media across the oxygen sensor. (See sales literature and instruction sheets for further information)

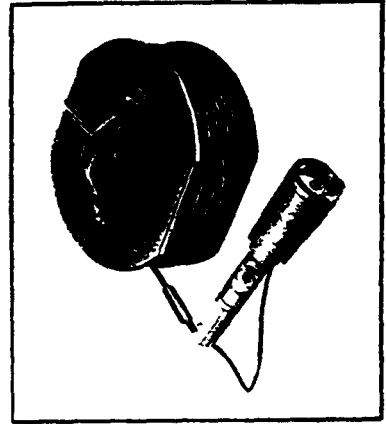


FIGURE 5

VI YSI 5721 Battery Pack and Charger

The YSI 5721 Battery Pack and Charger is offered as an accessory to operate either the YSI 5791A or 5795A Submersible Stirrer when the stirrer is used in conjunction with the YSI Model 57 Oxygen Meter. The YSI 5721 can be purchased with the YSI Model 57 or installed at a later time (See sales literature and instruction sheet for further information)

OPERATING PROCEDURES

I Preparing the Probe

All YSI 5700 Series Probes have similar sensors and should be cared for in the same manner. They are precision devices relying on good treatment if high accuracy measurements are to be made. Prepare the probes as follows (See Figure 6):

ALL PROBES ARE SHIPPED DRY — YOU MUST FOLLOW THESE INSTRUCTIONS

- 1 Prepare the electrolyte by dissolving the KCl crystals in the dropper bottle with distilled water. Fill the bottle to the top.
- 2 Unscrew the sensor guard from the probe (YSI 5739 only) and then remove the O' ring and membrane. Thoroughly rinse the sensor with KCl solution.
- 3 Fill the probe with electrolyte as follows:

A Grasp the probe in your left hand. When preparing the YSI 5739 probe the pressure compensating vent should be to the right. Successively fill the sensor body with electrolyte while pumping the diaphragm with the eraser end of a pencil or similar soft, blunt tool. Continue filling and pumping until no more air bubbles appear. (With practice you can hold the probe and pump with one hand while filling with the other.) When preparing the YSI 5720A and 5750 probes simply fill the sensor body until no more air bubbles appear.

B Secure a membrane under your left thumb. Add more electrolyte to the probe until a large meniscus completely covers the gold cathode. NOTE: Handle membrane material with care, keeping it clean and dust free, touching it only at the ends.

C With the thumb and forefinger of your other hand, grasp the free end of the membrane.

D Using a continuous motion, stretch the membrane UP, OVER, and DOWN the other side of the sensor. Stretching forms the membrane to the contour of the probe.

E Secure the end of the membrane under the forefinger of the hand holding the probe.

F Roll the "O" ring over the end of the probe. There should be no wrinkles in the membrane or trapped air bubbles. Some wrinkles may be removed by lightly tugging on the edges of the membrane beyond the O ring.

G Trim off excess membrane with scissors or sharp knife. Check that the stainless steel temperature sensor is not covered by excess membrane.

Shake off excess KCl and reinstall the sensor guard.

4 A bottomless plastic bottle is provided with the YSI 5739 probe for convenient storage. Place a small piece of moist towel or sponge in the bottle and insert the probe into the open end. This keeps the electrolyte from drying out. The YSI 5720A and 5750 probes can be stored in a B O D bottle containing about 1" water.

Membranes will last indefinitely, depending on usage. Average replacement is 2-4 weeks. However, should the electrolyte be allowed to evaporate and an excessive amount of bubbles form under the membrane, or the membrane become damaged, thoroughly flush the reservoir with KCl and install a new membrane.

7. Also replace the membrane if erratic readings are observed or calibration is not stable.

8. "Home brew" electrolyte can be prepared by making a saturated solution of reagent grade KCl and distilled water, and then diluting the solution to half strength with distilled water. Adding two drops of Kodak Photo Flo per 100 ml of solution assures good wetting of the sensor, but is not absolutely essential.

9. The gold cathode should always be bright and untarnished. If it is tarnished (which can result from contact with certain gases) or plated with silver (which can result from extended use with a loose or wrinkled membrane), return it to the factory for service or else clean it with the YSI 5680 Probe Reconditioning Kit. Never use chemicals or any abrasive other than that supplied with this kit.

10. It is also possible that the silver anode may become contaminated, which will prevent successful calibration. Try soaking the probe overnight in a 3% ammonia solution, rinse with deionized water, recharge with electrolyte, and install a new membrane. If still unable to calibrate, return the probe for service.

11. H_2S , SO_2 , Halogens, Neon, Nitrous Oxide and CO are interfering gases. If you suspect erroneous readings it may be necessary to determine if these are the cause. These gases have been tested for response:

100% Carbon Monoxide-Less than 1%	100% Helium-none
100% Carbon Dioxide-Around 1%	100% Nitrous Oxide-1/3 O_2 response
100% Hydrogen-Less than 1%	100% Ethylene-none
100% Chlorine-2/3 O_2 response	100% Nitric Oxide-1/3 O_2 response

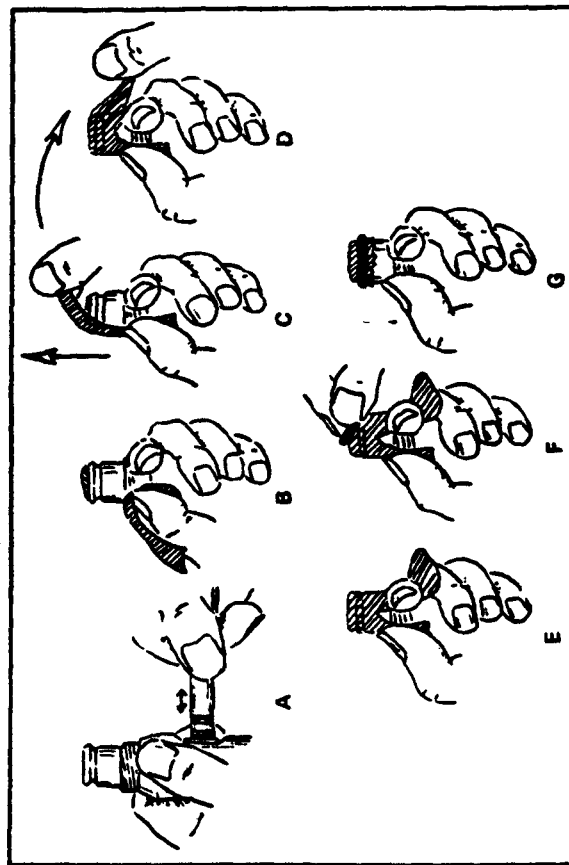


FIGURE 6

II Preparing Instrument

It is important that the instrument be placed in the intended operating position vertical, tilted or on its back — before it is prepared for use and calibrated (See Figure 7) Readjustment may be necessary when the instrument operating position is changed After preparing the probe proceed as follows

- 1 With switch in the OFF position adjust the meter pointer to Zero with the screw in the center of the meter panel Readjustment may be necessary if the instrument position is changed
- 2 Switch to RED LINE and adjust the RED LINE knob until the meter needle aligns with the red mark at the 31°C position
- 3 Switch to ZERO and adjust to zero with zero control knob
- 4 Attach the prepared probe to the PROBE connector of the instrument and adjust the retaining ring finger tight
- 5 Before calibrating allow 15 minutes for optimum probe stabilization Repolarize whenever the instrument has been OFF or the probe has been disconnected

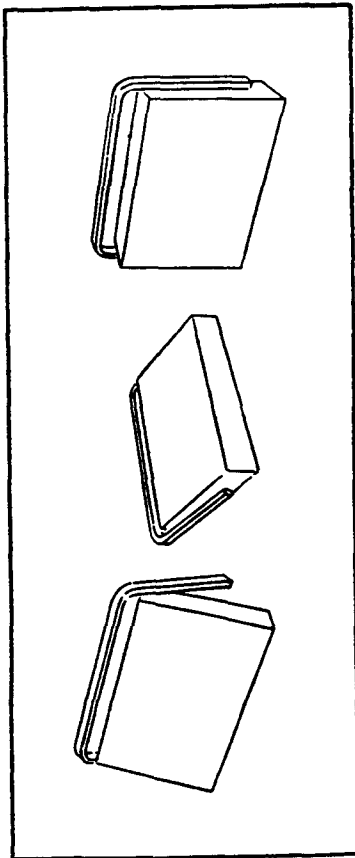


FIGURE 7

III Calibration

The operator has a choice of three calibration methods — Winkler Titration Saturated Water and Air Experience has shown that air calibration is quite reliable yet far simpler than the other two methods The three methods are described in the following paragraphs

Winkler Titration

- 1 Draw a volume of water from a common source and carefully divide into four samples Determine the oxygen in three samples using the Winkler Titration technique and average the three values If one of the values differs from the other 2 by more than 0.5 mg/l, discard that value and average the remaining two
- 2 Place the probe in the fourth sample and stir
- 3 Set the SALINITY control to zero or the appropriate salinity value of the sample
- 4 Switch to desired mg/l range and adjust the CALIBRATION control to the average value determined in Step 1 Allow the probe to remain in the sample for at least two minutes before setting the calibration value, and leave in the sample for an additional 2 minutes to verify stability Readjust if necessary

Saturated Water

- 1 Air saturate a volume of water (300-500cc) by aerating or stirring for at least 15 minutes at a relatively constant temperature
 - 2 Place the probe in the sample and stir Switch to TEMPERATURE Refer to Calibration Table I for the mg/l value corresponding to the temperature
 - 3 Determine local altitude or the true atmospheric pressure (note that true atmospheric pressure is as read on a mercury barometer Weather Bureau reporting of atmospheric pressure is corrected to sea level) Using Table II determine the correct factor for your pressure or altitude
 - 4 Multiply the mg/l value from Table I by the correction factor from Table II to determine the corrected calibration value for your conditions
- EXAMPLE** Assume temperature = 21°C and altitude = 1000 feet From Table I the calibration value for 21°C is 8.9 mg/l From Table II the correction factor for 1000 feet is about 0.96 The corrected calibration value is $8.9 \text{ mg/l} \times 0.96 = 8.54 \text{ mg/l}$
- 5 Switch to an appropriate mg/l range, set the SALINITY knob to zero, and adjust the CALIBRATE knob while stirring until the meter reads the corrected calibration value from Step 4 Leave the probe in the sample for two minutes to verify calibration stability Readjust if necessary

Air Calibration

- 1 Place the probe in moist air BOD probes can be placed in partially filled (50 mL) BOD bottles Other probes can be placed in the YSI 5075A Calibration Chamber (refer to the following section describing calibration chamber) or the small storage bottle (the one with the hole in the bottom) along with a few drops of water The probe can also be wrapped loosely in a damp cloth taking care the cloth does not touch the membrane Wait approximately 10 minutes for temperature stabilization

- 2 Switch to TEMPERATURE and read Refer to Table I — Solubility of Oxygen in Fresh Water and determine calibration value

- 3 Determine altitude or atmospheric correction factor from Table II

- 4 Multiply the calibration value from Table I by the correction factor from Table II

EXAMPLE Assume temperature = 21°C and altitude = 1000 feet From Table I the calibration value for 21°C is 8.9 mg/l From Table II the correction factor for 1000 feet is about 0.96 Therefore, the corrected calibration value is $8.9 \text{ mg/l} \times 0.96 = 8.54 \text{ mg/l}$

- 5 Switch to the appropriate mg/l range, set the SALINITY knob to zero and adjust the CALIBRATE knob until the meter reads the correct calibration value from Step 4 Wait two minutes to verify calibration stability Readjust if necessary

The probe is now calibrated and should hold this calibration value for many measurements Calibration can be disturbed by physical shock touching the membrane or drying out of the electrolyte Check calibration after each series of measurements and in time you will develop a realistic schedule for recalibration For best results when not in use follow the storage procedures recommended for the various probes described under OXYGEN PROBES AND EQUIPMENT This will reduce drying out and the need to change membranes

Calibration Chamber

The YSI 5075A Calibration Chamber is an accessory that helps obtain optimum calibration in the field and is also a useful tool for measuring at shallow depths (less than 4').

As shown in Figure (A), it consists of a 4-1/2 foot stainless steel tube (1) attached to the calibration chamber (2), the measuring ring (3), and two stoppers (4) and (5).

For calibration, insert the solid stopper (4) in the bottom of the calibration chamber (2). Push the oxygen probe (6) through the hollow stopper (5) as shown in Figure (B). Place the probe in the measuring ring, Figure (C), and immerse the probe in the sample to be measured for five minutes to thermally equilibrate the probe. Quickly transfer the probe to the calibration chamber (5) draining excess water from the chamber and shaking any excess droplets from the probe membrane. For maximum accuracy, wet the inside of the calibration chamber with fresh water. This creates a 100% relative humidity environment for calibration. Place the chamber in the sample for an additional five minutes for final thermal equilibrium. Calibrate the probe as described in the air-calibration procedure. Keep the handle above water at all times.

After calibration, return the probe to the measurement ring for shallow measurements. Move the probe up and down, or horizontally, approximately one foot a second while measuring. In rapidly flowing streams (greater than 5/sec.) install the probe in the measuring ring with the pressure compensating diaphragm towards the chamber.

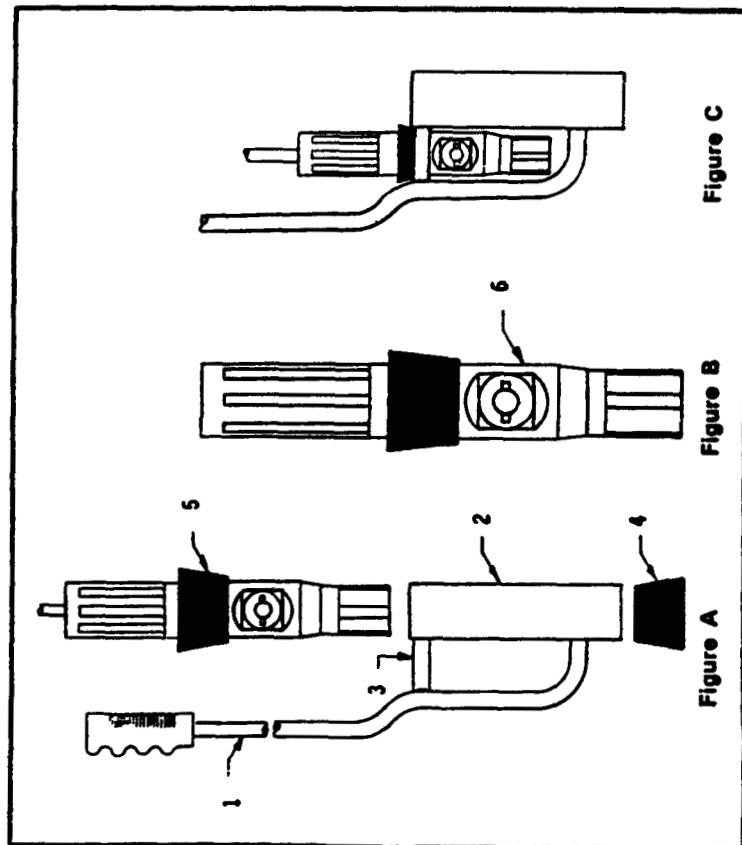


FIGURE 8

Dissolved Oxygen Measurement

With the instrument prepared for use and the probe calibrated place probe in the sample to be measured and provide stirring.

1. Stirring for the 5739 Probe can best be accomplished with a YSI submersible stirrer. Turn the STIRRER knob ON. If the submersible stirrer is not used, provide manual stirring by raising and lowering the probe about 1 ft per second. If the 5075A Calibration Chamber is used, the entire chamber may be moved up and down in the water at about 1 ft per second.
2. The YSI 5720A has a built-in power driven stirrer.
3. With the YSI 5750 sample stirring must be accomplished by other means such as with the use of a magnetic stirring bar.
4. Adjust the SALINITY knob to the salinity of the sample.
5. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen. Read dissolved oxygen.

V Calibration Tables

Table I shows the amount of oxygen in mg/l that is dissolved in air saturated fresh water at sea level (760 mmHg atmospheric pressure) as temperature varies from 0° to 45°C.

Table I — Solubility of Oxygen in Fresh Water

Temperature °C	mg/l Dissolved Oxygen	Temperature °C	mg/l Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source Derived from 15th Edition "Standard Methods for the Examination of Water and Wastewater"

Table II shows the correction factor that should be used to correct the calibration value for the effects of atmospheric pressure or altitude. Find true atmospheric pressure in the left hand column and read across to the right hand column to determine the correction factor. (Note that true atmospheric pressure is as read on a barometer. Weather Bureau reporting of atmospheric pressure is corrected to sea level.) If atmospheric pressure is unknown, the local altitude may be substituted. Select the altitude in the center column and read across to the right hand column for the correction factor.

Table II — Altitude Correction Factor

Atmospheric Pressure mmHg	or	Equivalent Altitude Ft	Correction Factor
775		540	1.02
760		0	1.00
745		542	.98
730		1094	.96
714		1688	.94
699		2274	.92
684		2864	.90
669		3466	.88
654		4082	.86
638		4756	.84
623		5403	.82
608		6065	.80
593		6744	.78
578		7440	.76
562		8204	.74
547		8939	.72
532		9694	.70
517		10472	.68
502		11273	.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

VI. HIGH SENSITIVITY MEMBRANE

Use of high sensitivity 0005" membranes (YSI 5776) in place of standard 001" membranes (YSI 5775) when measurements are to be made consistently at low temperatures (less than 15°C). Calibration and readings will be made just as if the standard YSI 5775 membrane was being used.

The YSI 5776 High Sensitivity Membrane can also be used in certain situations to increase sensitivity at temperatures above 15°C. The ranges thus become 0-2.5, 0-5 and 0-10 mg/l. When calibration with high sensitivity membranes is attempted at temperatures greater than 15°C the selector switch must be set to 0-20 mg/l. Multiply the calculated calibration value by 2. For example at 21°C and 1000 ft altitude the calibration value would be 8.6 x 2 or 17.2. Remember the 0-5, 0-10 and 0-20 mg/l ranges are now 0-2.5, 0-5 and 0-10 mg/l, and all mg/l readings must be divided by 2 for a final reading. When operating in this manner accuracy will be degraded slightly.

RECORDER OUTPUT

Output at full scale is 114 to 136 mV

Use a 50K or higher input impedance recorder and operate it with the terminals ungrounded.

Many recorders have an adjustable full scale sensitivity feature. When using this type use the 100 mV range and adjust the full scale (span range control, sensitivity etc.) control to give full scale chart deflection with full scale oxygen meter deflection. Refer to the recorder instructions. For recorders without this feature a simple driver network as shown below can be constructed. This is adequate to adjust the signal for full scale chart and meter deflection on the 100 mV fixed range recorders.

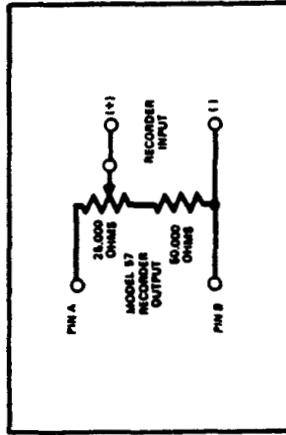


FIGURE 9

Recorder Output Plug

The YSI Model 57 is supplied with the necessary parts to construct a waterproof recorder plug for the YSI Model 57 Dissolved Oxygen Meter. The cable and potting materials are not included. (See Figure 10).

General purpose epoxy potting materials of medium viscosity and moderate cure rate are recommended. The two tube kits available in hardware stores are satisfactory.

- 1 Prepare the cable end by stripping back 3/16 (5MM) of insulation. Tin the ends with rosin core solder. If polarity is important, pin "A" is the (+) terminal.
- 2 Disassemble the connector pieces and slide the mold, ring, extension, and coupling nut over the cable. Solder the leads to the appropriate connector pins with rosin core solder.
- 3 Check all connections. The two leads should show electrical continuity to the pins and should not contact the body or each other.
- 4 Re-assemble the pieces and hold the connector upright. Pour the epoxy mix into the plastic mold until full. Refill as the epoxy settles.
- 5 After the epoxy cures the plastic mold may be removed with pliers or knife.

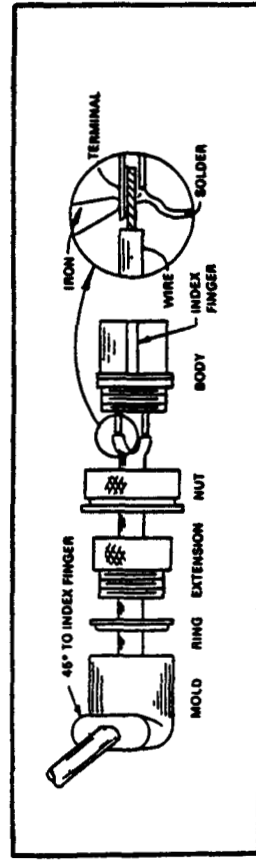


FIGURE 10

DISCUSSION OF MEASUREMENT ERRORS

There are three basic types of errors which can occur. Type I errors are related to limitations of the instrument design and tolerances of the instrument components. These are chiefly the meter linearity and resistor tolerances. Type II errors are due to basic probe accuracy tolerances, chiefly background signal probe linearity, and variations in membrane temperature coefficient. Type III errors are related to the operator's ability to determine the conditions at the time of calibration. If calibration is performed against more accurately known conditions, Type III errors are appropriately reduced.

Individual Sources of Error

This description of sources of error can be used to attach a confidence to any particular reading of dissolved oxygen. The particular example given is for a near extreme set of conditions. As a generality, overall error is diminished when the probe and instrument are calibrated under conditions of temperature and dissolved oxygen which closely match the sample temperature and dissolved oxygen.

Type I

A Is the error due to the meter linearity

Error = $\pm 1\%$ of full scale of the measurement range

B Is the error due to tolerances in the instrument when transferring a reading from one range to another

Error = $\pm 1\%$ of the meter reading if the reading is taken on a range one range away from the calibration range

Error = $\pm 2\%$ of the meter reading if the reading is taken on a range two ranges away from the calibration range

C Is the error due to the design and components of the instrument salinity compensation circuit

Error = $\pm 2.5\%$ of the meter reading $\times \frac{\text{sample salinity ppt}}{40 \text{ ppt salinity}}$

Type II

A errors are due to probe background current

Error = $0.5\% \left(\frac{\text{meter reading mg/l}}{1 - \text{Calib value}} \right) \times \text{Calib value, mg/l}$

B errors are due to the probe non-linearity

Error = 0.3% of reading

C error is caused by variability in the probe membrane temperature coefficient

Error = zero if readings are taken at the calibration temperature

Error = $\pm 1\%$ of meter reading if readings are taken with 5°C of the calibration temperature

Error = $\pm 3\%$ of meter reading all other conditions

Type III

A errors are due to the accuracy of the instrument thermometer when used to measure the exact probe temperature during calibration

Error = $\pm 1.5\%$ of reading

B errors are due to the assumption of mean, barometric pressure

Daily variation is usually less than 1.7%

Error = $\pm 1.7\%$ of reading

errors assume an ability to estimate altitude to within ± 500 ft when putting the altitude correction factor

Error = 1.8% of reading

D errors consider the possibility of only 50% relative humidity when calibrating the probe. If the actual relative humidity is 50% instead of 100% the errors will be as follows

Calibration Temperature \pm C	Error in Percent of Reading
0	(-) 0.3
10	(-) 0.6
20	(-) 1.15
30	(-) 2.11
40	(-) 3.60

Example of a Typical Error Calculation

The example given presumes the air calibration technique. If calibration is done with air saturated water, the relative humidity consideration (III-D) is eliminated. If the Winkler calibration method is used, Type III errors are deleted and replaced by the uncertainty attributable to the overall Winkler determination. Data: Instrument calibrated at 25°C , elevation estimated at 2000 feet ± 500 feet, normal barometric pressure presumed calibrated on $0-10$ mg/l scale at 7.8 mg/l. Readings taken on $0-5$ mg/l range at 4.5 mg/l, temperature 20°C . Salinity of 20 ppt

Type	Description	Calculations	Error mg/l
IA	Linearity	= 01 x 4 5 mg/l	045
IB	Range Change	= 01 x 4 5 mg/l	045
IC	Salinity	= 025 x 4 5 mg/l x 40 ppt	056
IIA	Probe Background	= 005 x $\left(1 - \frac{4.5 \text{ mg/l}}{7.8 \text{ mg/l}}\right) \times 7.8 \text{ mg/l}$	016
IIB	Probe Linearity	= 003 x 4 5 mg/l	014
IIC	Temp Compensation	= 01 x 4 5 mg/l	045
IIIA	Temp Measurement	= 015 x 4 5 mg/l	068
IIIB	Pressure	= 017 x 4 5 mg/l	076
IIIC	Altitude	= 018 x 4 5 mg/l	081
IIID	RH	= 016 x 4 5 mg/l	072
Maximum Possible Error = 518 mg/l			± 259
Probable Error			

Considering a statistical treatment of the probable error at any time for any instrument, it is likely that the actual error in any measurement will be about $1/2$ of the possible error. In this case the probable error is about ± 2.6 mg/l out of a reading of 4.5 mg/l or 5.8% of the reading.

INSTRUMENT CASE

The instrument case is water resistant when properly closed. As a precaution against damaged gaskets or loose fittings, the instrument case should be opened and inspected for moisture whenever the instrument has been subjected to immersion or heavy spray. The instrument case is opened by removing the screws on the rear cover and lifting the cover off.

INSTRUMENT BATTERIES

The instrument batteries are two "C" size carbon-zinc cells located inside the instrument on the meter end. These should be replaced when the RED LINE knob is at its extreme adjustment or at least annually. The amount of remaining adjustment is an indication of the battery condition. The batteries are replaced by removing the screws on the rear cover of the instrument and removing the two batteries at the end of the instrument near the meter. When installing the new batteries the plus (+) end fits into the red washer on the battery holder. (See Figure 11)

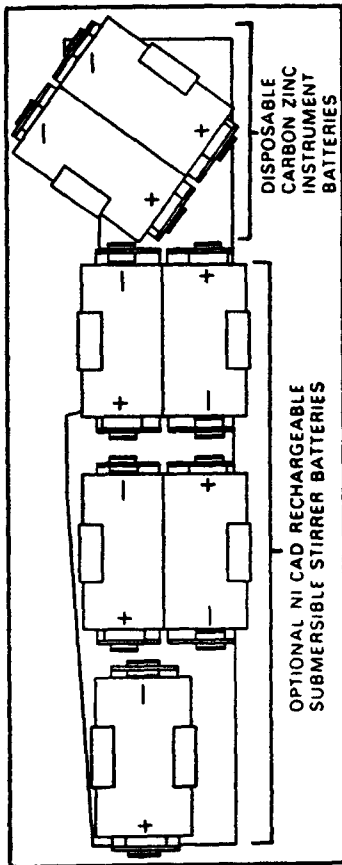


FIGURE 11

WARRANTY AND REPAIR

All YSI products carry a one-year warranty on workmanship and parts, exclusive of batteries. Damage through accident, misuse, or tampering will be repaired at a nominal charge, if possible, when the item is returned to the factory or to an authorized YSI dealer.

If you are experiencing difficulty with any YSI product, it may be returned for repair, even if the warranty has expired. YSI maintains complete facilities for prompt servicing for all YSI products.

PRODUCT SERVICE DEPARTMENT

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